

Protocol for evaluating the performance of Near-Infrared spectrometers

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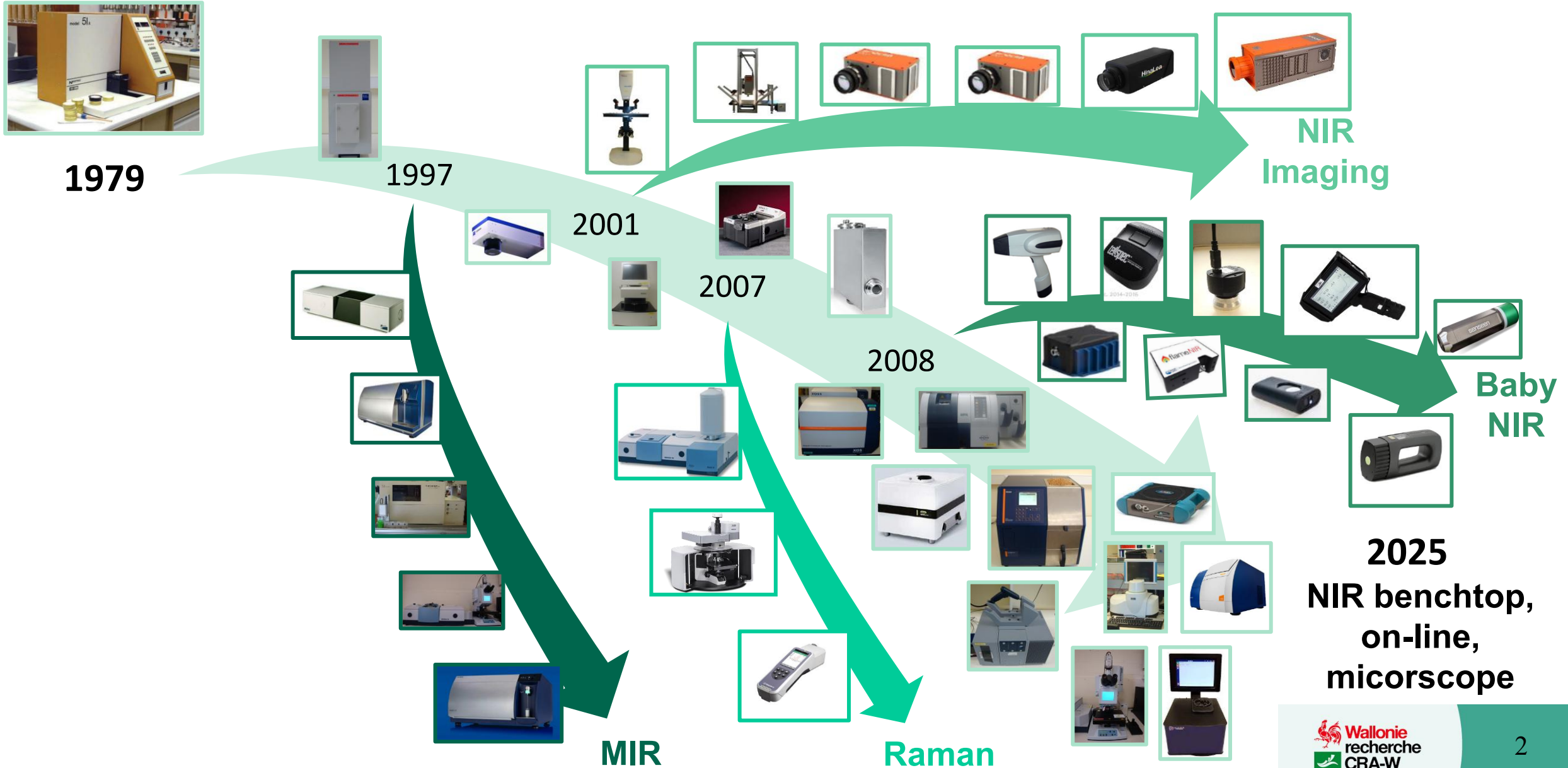
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History of Spectroscopy at CRA-W



How to evaluate the performances of new NIR spectrometers

How to know what to expect from new NIR instruments ?

→ **Applied protocol for evaluating the performances**

Evaluation of:

- Short-term repeatability (few seconds)
- Medium-term repeatability (minutes to hours)
- Stability over time (long term (days to weeks))
- Signal-to-noise ratio, RMS, ...
- Ability to measure different matrices (whole grain, soil, etc.)
- Transferability between instruments



Minimize sources of exogenous variation (controlled temperature $22 \pm 3^{\circ}\text{C}$, light, ...)

Use manufacturer's measurement configuration

→ **Evaluation in laboratory conditions**

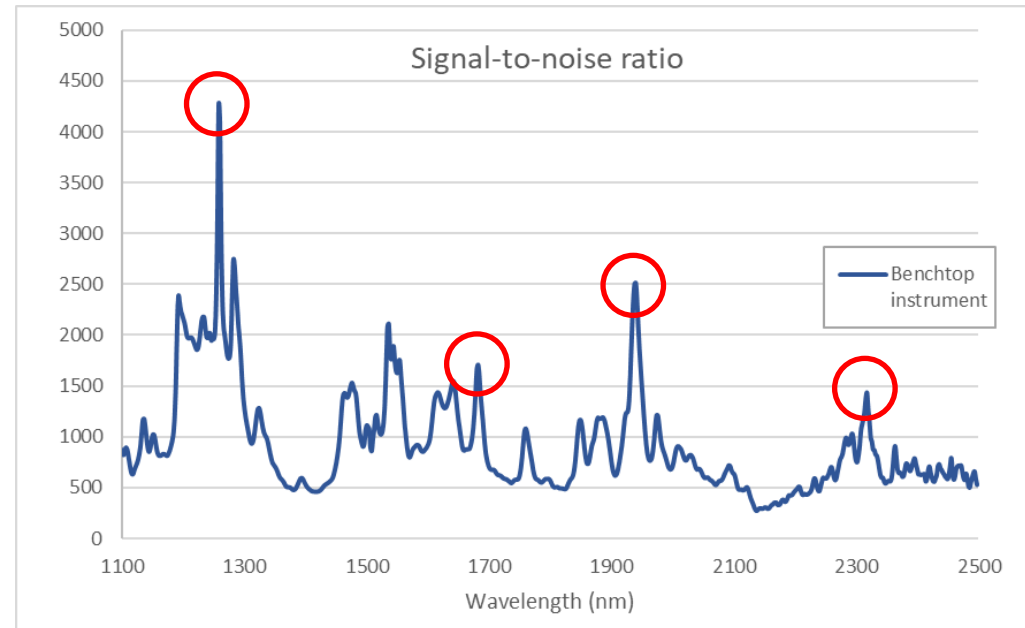
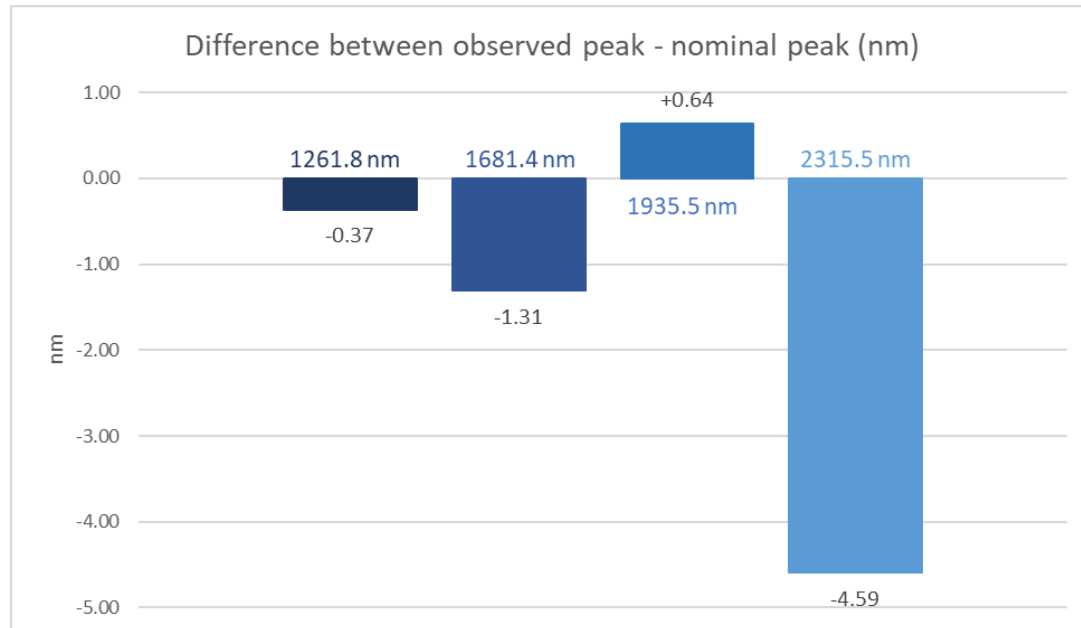
Evaluation of accuracy and short-term repeatability

Very homogeneous rare earth that posses 7 nominal peaks in NIR (nm)



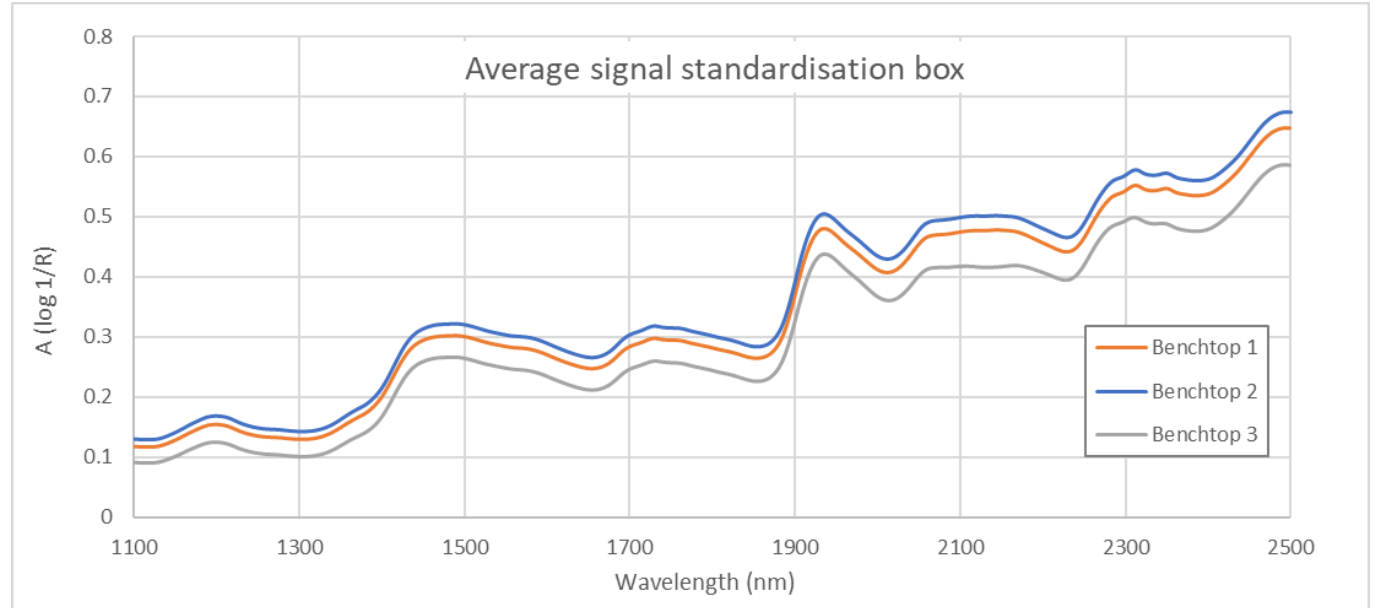
655.1	886.5	975.5	1261.8	1681.4	1935.5	2313.5
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- 10 consecutive measurements
 - Peak position
 - Signal-to-noise-ratio
 - Short-term repeatability (RMS)



Evaluation of medium-term repeatability, transferability, ...

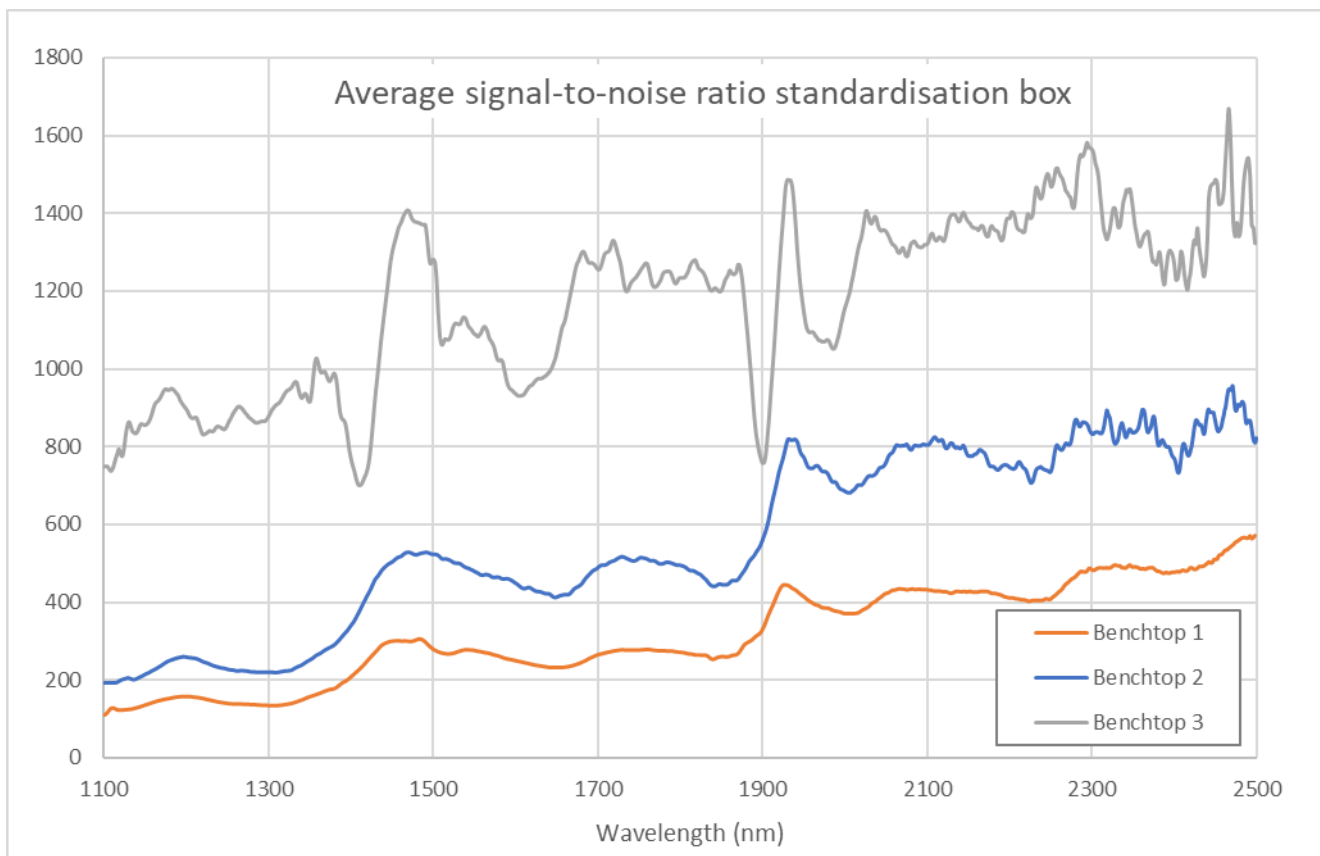
Standardization box containing 40 sealed products with varying spectra signature, used for powder standardization



- Measurement in non-consecutive duplicates
- Reference (blank) taken every 10 measurements
- Signal-to-noise ratio
- Medium-term repeatability (RMS)
- Transferability



Measurement of standardization box



Medium-term repeatability

Aiming for an average RMS < 1000 for these sealed cups on benchtop NIR spectrometers

Sample	Benchtop 1	Benchtop 2	Benchtop 3
# 1	98	712	102
# 2	176	737	460
# 3	126	776	129
# 4	206	524	108
# 5	131	581	101
# 6	177	620	358
...			
# 39	126	862	109
# 40	452	524	101
Average	542	363	149

Results show that all three benchtop spectrometers are operating within the desired range



Interpreting performance differences

- For the same samples, the RMS was sometimes higher on one instrument than another, indicating significant variability
- Variability within the same instrument for the same standardization box

Average RMS XDS CRA-W

Year	Average RMS
2014	237
2015	160
2016	143
2017	383
2018	240
2019	256
2020	167
2021	398
2022	245



9 * XDS CRA-W

- Mean: **248**
- Standard deviation: **91**
- confidence interval (95 %; $\pm 1.96 * SD$): **69-426**

Average RMS XDS Requasud Network 2022

Laboratory	Average RMS
1	245
2	202
3	351
4	187
5	254
6	538
7	402
8	459
9	727
10	218



10 XDS Requasud

- Mean: **358**
- Standard deviation: **176**
- confidence interval (95 %; $\pm 1.96 * SD$): **14-702**

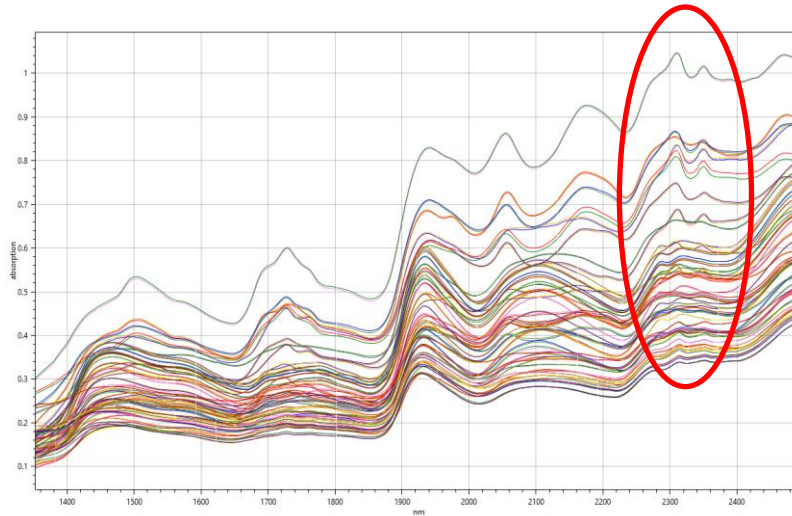
Interpret with caution: not about comparing or ranking, but verifying whether performance is acceptable



Transferability between instruments

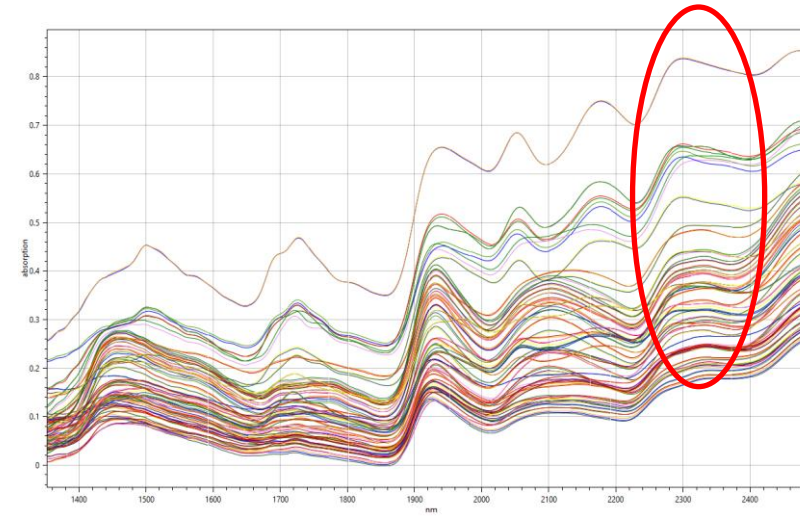
- Is it possible to transfer historical database to new instruments (PDS standardization) ?

Benchtop spectrometer

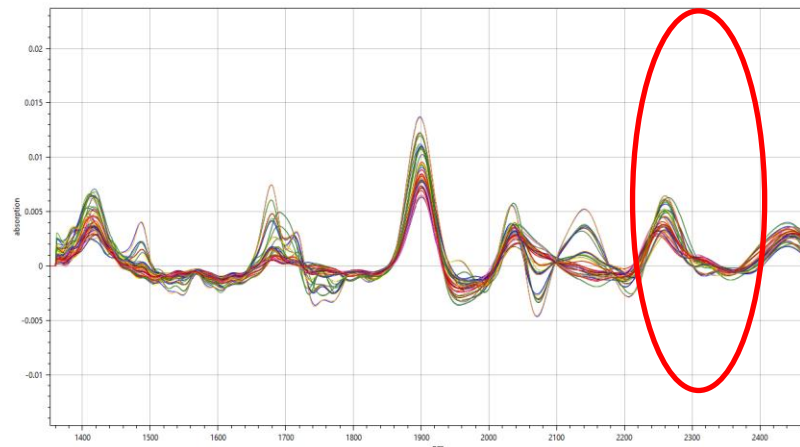
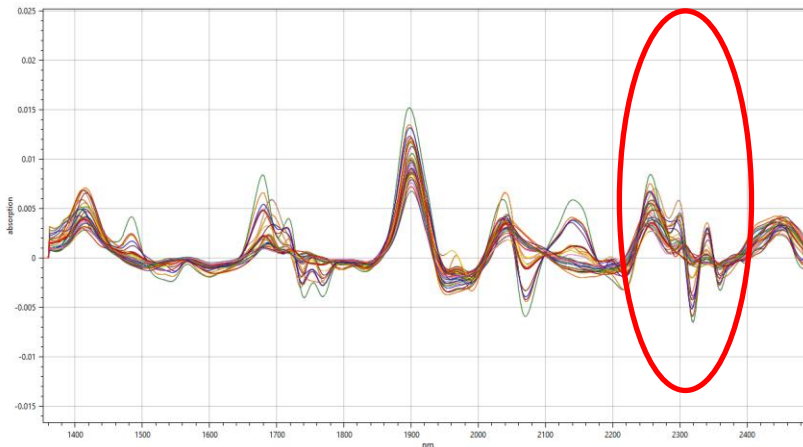


Raw spectra

Handheld spectrometer



SG Derivative
(1,4,41)



**Database
transfer from
benchtop to
handheld device
challenging**

Ability to measure different matrices (wheat, soil)



20 wheat samples with reference values (moisture, protein)

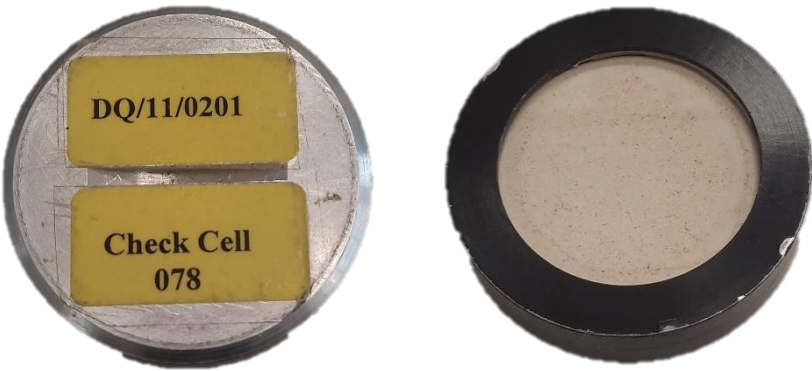
- Measurement in non-consecutive duplicates (double filling)
- Reference (blank) taken every 10 measurements
- Signal-to-noise ratio, RMS for less heterogeneous sample
- Ability to make a model (max 3 latent variables)

	LV	N	Mean	SD	SEC	RSQ	SECV	RPD
Benchtop 1								
Moisture (%)	3	20	13.50	0.82	0.14	0.97	0.18	4.59
Protein 5.7 (%)	3	20	11.93	1.74	0.41	0.95	0.69	2.51
Benchtop 2								
Moisture (%)	3	20	13.50	0.82	0.12	0.98	0.16	5.26
Protein 5.7 (%)	3	20	11.93	1.74	0.29	0.97	0.47	3.74
Benchtop 3								
Moisture (%)	3	20	13.50	0.82	0.13	0.98	0.17	4.94
Protein 5.7 (%)	3	20	11.93	1.74	0.37	0.96	0.56	3.09

Use of measuring accessories

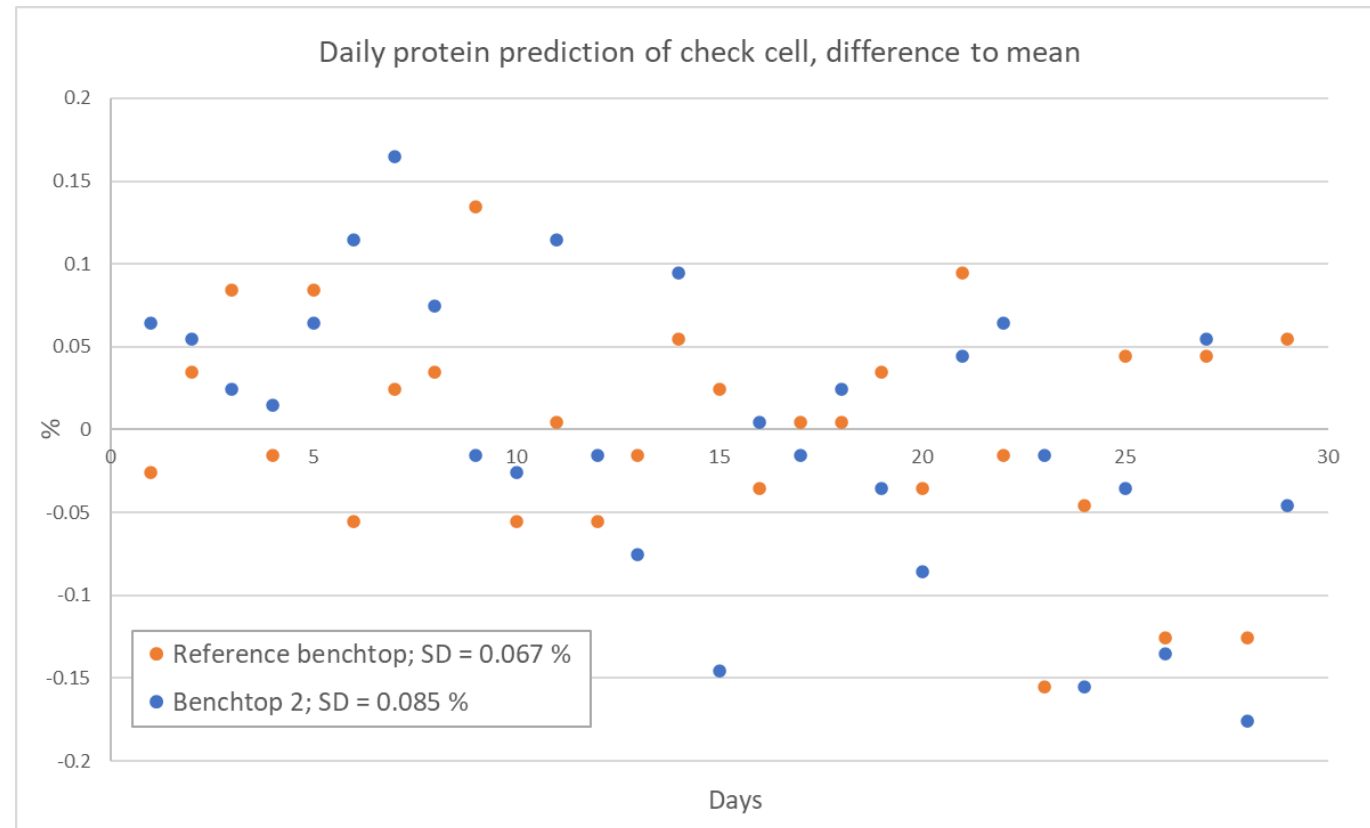
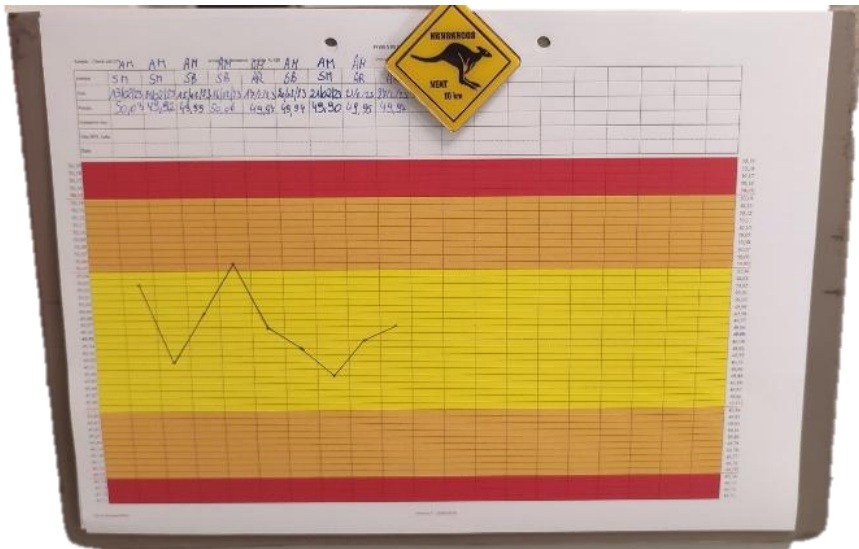
Can help to determine the optimal number of repetition for less heterogeneous samples

Stability in time: Check cell



Sealed cup with animal feed

- Measured once a day during the duration of the protocol
- Evaluation of instrument stability over time (protein prediction)



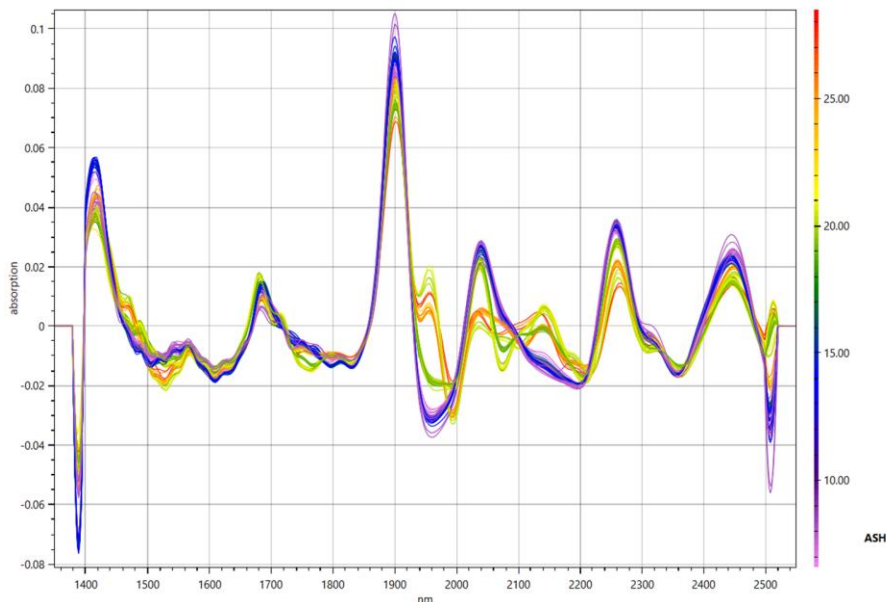
Ability to perform models



Set of 80 sealed feed sample cells with reference values

- Measured over a period of 4 weeks (one box per week)
- Medium-term repeatability via RMS
- Device stability over time
- Model development using different cross-validation strategies
 - Leave-one-week (box) –out (**LWO**) (n=20)
 - Leave-one-out (**LOO**) (n=1)
 - 4 random groups (**4GR**) (n=20)

80 SGol-derived feed spectra



	CV	SD	SECV	RPD
FAT	LWO	1.50	0.78	1.92
(%)	LOO	1.50	0.73	2.04
	4GR	1.50	0.83	1.81
FIBER	LWO	3.57	1.31	2.73
(%)	LOO	3.57	1.26	2.83
	4GR	3.57	1.33	2.69
ASH	LWO	5.72	1.82	3.15
(%)	LOO	5.72	1.72	3.33
	4GR	5.72	1.85	3.1

Conclusion

- Well-known and characterized samples that help to quickly understand how the instrument operates and to identify its most suitable applications
- Applied strict protocol when you want to challenge and evaluate an NIR instrument
 - Controlled laboratory conditions even for handheld devices typically intended for field use
- Know well and discuss with instrument provider
 - Adapt to the device and its measuring accessories (measurements, replicates, ...)
 - Focus on specific spectral ranges

Thank you for your attention



1979

1990 - Brussels, Belgium

1992 - Haugesund, Norway

1995 - Montreal, Canada

1999 - Verona, Italy

2003 - Cordoba, Spain

2007 - Umeå-Vaasa, Sweden/Finland

2011 - Cape Town, South Africa

2015 - Foz do Iguaçu, Brazil

2019 - Gold Coast, Australia

2023 - Innsbruck, Austria

2027 - Nagoya, Japan

1989 - Tsukuba, Japan

1991 - Aberdeen, Scotland

1994 - Australia

1997 - Essen, Germany

2001 - Kyongju, South Korea

2005 - Auckland, New Zealand

2009 - Bangkok, Thailand

2013 - La Grande-Motte, France

2017 - Copenhagen, Denmark

2021 - Beijing, China

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REGISTRATION

<https://gqr.sh/bbn2>



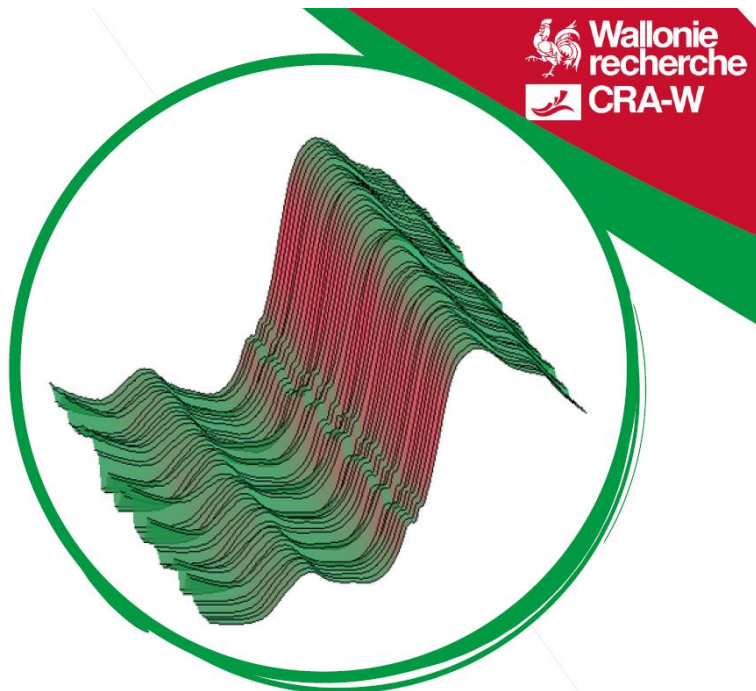
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- Juan Antonio
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